TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

METHOD SUMMARY & DATA QUALITY OBJECTIVES

TestAmerica West Sacramento
SW8290: Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography / High Resolution Mass Spectrometry (HRGC/HRMS)

This method provides instrument and extraction procedures for the detection and quantitation of PCDDs (tetra through octa-chlorinated homologues) and PCDFs (tetra through octa-chlorinated homologues) in a variety of sample matrices in part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations.

Method SW8290 is used to detect dioxins and furans in variety of matrices and uses additional quality controls to allow more sophisticated determinations of detection limits and target analyte concentrations than other routine GC and GC/MS methods.

Method SW8290 requires that isotopically labeled analogs of target analytes be spiked into each sample before extraction, and uses nine ^{13}C labeled analogs, one furan and one dioxin at each chlorination level. 13C-OCDF is not used as an internal standard due to its potential interference with OCDD and 13C-1,2,3,7,8,9-HxCDD is used as a recovery standard. By adding a known amount of labeled compounds to every sample prior to extraction, correction for recovery of the target analytes can be made because the target analytes and their labeled analog exhibit similar effects upon extraction, cleanup, concentration, and gas chromatography. Target analytes are quantitated relative to the labeled analog and therefore their calculated concentration compensates for extraction and cleanup efficiencies.

A batch specific LCS (Laboratory Control Sample) is not required by Method 8290, however, TestAmerica West Sacramento still analyzes an LCS at a frequency of 1 per batch of 20 samples as an ongoing system and standard check. The target analyte concentrations for the LCS are given in Table 2. Sample matrix spikes and/or spike duplicates are performed only at client request. The spike concentrations are nominal values based on a full volume sample preparation (1000 mls for liquids and 10 grams for solids). If less than a full volume of sample is prepared due to sample matrix, sample availability, or method requirements, the spike amount will remain constant and therefore the spike concentrations will vary. See Table 2 through Table 4 for specific QC control and corrective action measures.

Detection Limits and Reporting Limits:

TestAmerica West Sacramento's Method SW8290 provides customizable options to report detection limits and/or reporting limits.

- Reporting Limit (RL) - When target analytes meet method identification criteria and are free of interferences, they are reported down to the lowest calibration standard concentration (see reporting limits in Table 1). Data can be reported to the RL without the use of qualification if required.
- Estimated Detection Limit (EDL) - For each analyte not detected, an EDL can be reported. The sample specific EDL is an estimate of the concentration of a given analyte that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, etc. Because of the toxicological significance of dioxins, the EDL value can be reported for non-detected chemicals rather than reporting the reporting limit (RL). Any analyte with a peak greater than 2.5 times the noise and meets all qualitative requirements but less than the RL would be reported with a "J" flag.
- Method Detection Limit (MDL) – Qualitatively confirmed analytes are reported as "estimated" down to the statistically derived MDL to denote the less certain quantitation and the value is qualified with a "J" flag. Any peak with a calculated concentration below the MDL is reported as "not detected" with no further qualification.

Second column confirmation will be performed only for 2,3,7,8-TCDF positives as per the convention selected..

Toxicity Equivalence Factors (TEFs)

As per client request, the 2,3,7,8-TCDD toxicity equivalence can be calculated in accordance with the procedures given in one of three different formats:

- TEF values cited in the U.S. Environmental Protection Agency, (1989) "Interim procedures for estimating risks associated with exposures to mixtures of chlorinated dibenzo-p-dioxins and –dibenzofurans (CDDs and CDFs) and 1989 update. U.S. Environmental Protection Agency, Risk Assessment forum, Washington DC; (EPA 625/3-89/016)."
- "WHO TEFs for human risk assessment based on the conclusions of the World Health Organization meeting in Stockholm, Sweden, 15-18, June 1997 (Van den Berg et aL, 1998)."
- "WHO TEFs for human risk assessment based on the conclusions of the World Health Organization meeting in Geneva, Switzerland, June 2005."

TEFs are assigned to each 2,3,7,8-substituted PCDDs/PCDFs in order to relate their toxicity to that of 2,3,7,8-TCDD. See Table 6 for the factors used to calculate TEFs. Note that EDL and detection limit values are not normally included in the TEQ adjusted concentration.

Uniform Federal Policy for Quality Assurance Project Plan (UFP-QAPP) Worksheets

UFP – QAPP Worksheets for Method 8290 pre-filled in with laboratory specific information are available upon request. Available tables include:

- Table 12 – Measurement Performance Criteria Table (Field QC and Laboratory QC Samples).
- Table 15 – Reference Limits and Evaluation Table
- Table 19 – Analytical SOP Requirements Table
- Table 23 – Analytical SOP References Table
- Table 24 – Analytical Instrument Calibration Table
- Table 25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
- Table 28 – Laboratory QC Samples Table
- Table 30 – Analytical Services Table

All tables are available in Microsoft Excel format for easy import into your proposal. Please ask your Project Manager for details.

TABLE 1 REPORTING LIMITS (RLs)
Based on Lower Calibration Limits
Method 8290 – TestAmerica

Lower detection limits are achievable using the estimated detection limit option.

Analyte	Water[1] (pg/L) RL	Soil/Sediment/Tissue[2] (pg/g) RL	Waste[3] (pg/g) RL
Dioxins			
2,3,7,8-TCDD	10	1.0	100
1,2,3,7,8-PeCDD	50	5.0	500
1,2,3,4,7,8-HxCDD	50	5.0	500
1,2,3,6,7,8-HxCDD	50	5.0	500
1,2,3,7,8,9-HxCDD	50	5.0	500
1,2,3,4,6,7,8-HpCDD	50	5.0	500
OCDD	100	10	1000
Furans			
2,3,7,8-TCDF	10	1.0	100
1,2,3,7,8-PeCDF	50	5.0	500
2,3,4,7,8-PeCDF	50	5.0	500
1,2,3,4,7,8-HxCDF	50	5.0	500
1,2,3,6,7,8-HxCDF	50	5.0	500
1,2,3,7,8,9-HxCDF	50	5.0	500
2,3,4,6,7,8-HxCDF	50	5.0	500
1,2,3,4,6,7,8-HpCDF	50	5.0	500
1,2,3,4,7,8,9-HpCDF	50	5.0	500
OCDF	100	10	1000

Note: “Totals” values are available upon client request.

[1] Based upon a 1.0 liter sample aliquot. Sensitivity of the method depends on the level of interferences rather than instrumental limitations.

[2] Based upon a 10.0 gram sample aliquot. Maximum RL for samples “as received”. Correction for moisture content may raise reporting limits above these levels.

[3] Based upon a 0.1 gram sample aliquot. Maximum RL for samples “as received”. Correction for moisture content may raise reporting limits above these levels. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

TABLE 2 CONTROL LIMITS FOR LABORATORY CONTROL SAMPLES (LCS), MATRIX SPIKES and MATRIX SPIKE DUPLICATES
Method 8290 – TestAmerica

Target Compound	LCS/MS/MSD Control Limits (Soil/Sediment)				LCS/MS/MSD Control Limits (Water)			
	AMT (pg/g)	Lower Control Limit	Upper Control Limit	RPD	AMT (pg/L)	Lower Control Limit	Upper Control Limit	RPD
Dioxins								
2,3,7,8-TCDD	20	77	133	20	200	71	128	20
1,2,3,7,8-PeCDD	100	74	145	20	1000	74	139	20
1,2,3,4,7,8-HxCDD	100	68	146	20	1000	65	144	20
1,2,3,6,7,8-HxCDD	100	79	141	20	1000	73	142	20
1,2,3,7,8,9-HxCDD	100	68	139	20	1000	60	147	20
1,2,3,4,6,7,8-HpCDD	100	74	147	20	1000	79	137	20
OCDD	200	75	153	20	2000	71	147	20
Furans								
2,3,7,8-TCDF	20	80	146	20	200	75	142	20
1,2,3,7,8-PeCDF	100	84	143	20	1000	80	140	20
2,3,4,7,8-PeCDF	100	76	157	20	1000	71	144	20
1,2,3,4,7,8-HxCDF	100	78	141	20	1000	64	149	20
1,2,3,6,7,8-HxCDF	100	78	144	20	1000	56	161	20
2,3,4,6,7,8-HxCDF	100	73	157	20	1000	60	169	20
1,2,3,7,8,9-HxCDF	100	70	144	20	1000	53	163	20
1,2,3,4,6,7,8-HpCDF	100	79	143	20	1000	78	141	20
1,2,3,4,7,8,9-HpCDF	100	79	150	20	1000	80	146	20
OCDF	200	70	158	20	2000	76	147	20

Note:
Native compound limits are TestAmerica West Sacramento historical limits and are subject to change.
RPD limits are currently set to the method default of 20%.
Tissue and waste control limits are available upon request.

TABLE 3 CONTROL LIMITS FOR INTERNAL STANDARDS
Method 8290 – TestAmerica

Internal Standard Compound	Internal Standards Control Limits (Soil/Sediment)			Internal Standards Control Limits (Water)		
	AMT (pg/g)	Lower Control Limit	Upper Control Limit	AMT (pg/L)	Lower Control Limit	Upper Control Limit
Dioxins						
13C-2,3,7,8-TCDD	200	40	135	2000	40	135
13C-1,2,3,7,8-PeCDD	200	40	135	2000	40	135
13C-1,2,3,6,7,8-HxCDD	200	40	135	2000	40	135
13C-1,2,3,4,6,7,8-HpCDD	200	40	135	2000	40	135
13C-OCDD	400	40	135	4000	40	135
Furans						
13C-2,3,7,8-TCDF	200	40	135	2000	40	135
13C-1,2,3,7,8-PeCDF	200	40	135	2000	40	135
13C-1,2,3,4,7,8-HxCDF	200	40	135	2000	40	135
13C-1,2,3,4,6,7,8-HpCDF	200	40	135	2000	40	135

Note:
Method default control limits. Signal-to-noise is also evaluated for data acceptability. These labeled analytes are spiked into all samples. Tissue and waste control limits are available upon request.

TABLE 4 SUMMARY OF CALIBRATION PROCEDURES
Method 8290 – TestAmerica

Calibration	Frequency	Acceptance Criteria	Corrective Action
Tune using PFK.	Prior to sample analysis and at the end of the analytical sequence (no time limit for the ending PFK analysis).	Resolving power ≥10,000 at m/z=304.9824 & m/z=380.9760 ± 5 ppm of expected mass.	1) Retune instrument. 2) Reanalyze PFK. 3) End resolution acceptable "as is" – assess data for impact if resolution is less than 10,000 and narrate or reinject as necessary.
Column Performance Check Solution (CPSM). Solution includes the Window Defining Mix.	Prior to 12 hrs of sample analysis.	Used to set retention times of first and last eluters. CPSM must have ≤25% valley resolution for 2,3,7,8-TCDD	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM. 5) No corrective action is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.
(5 point ICAL) Multipoint calibration.	Initially and as required.	1) I.S. = %RSD<30% 2) Natives = %RSD<20% 3) Retention time must be within -1 to +3 seconds of labeled I.S. or 0.005 RRT units. 4) Ion ratios within Table 5 limits, and I.S. S/N ≥10:1 and Natives S/N ≥2.5:1	1) Evaluate system. 2) Recalibrate. 3) If all criteria are met except #4 (ratio), evaluate impact, narrate and report if no impact is found.
Daily Continuing Calibration Verification standard (CCV).	Once per 12 hours, prior to sample analysis and at the end of the analytical sequence (no time limit for the ending CCV).	1) %D of I.S. ≤30% from avg. RRF (ICAL). (Ending %D of I.S. ≤35% from avg. RRF). 2) %D of natives ≤20% from avg. RRF (ICAL). (Ending %D of natives ≤25% from avg. RRF). 3) Retention time must be within -1 to +3 seconds of labeled I.S. or 0.005 RRT units. 4) Ion ratios within Table 5 limits, and I.S. S/N ≥10:1 and Natives S/N ≥2.5:1 5)	1) Evaluate system. 2) Evaluate data for usability. 3) Reanalyze (CCAL). 4) Recalibrate (ICAL) as necessary.

TABLE 4 SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES
Method 8290 – TestAmerica

QC Element	Frequency	Acceptance Criteria	Corrective Action
Internal Standards	Every sample, method blank, and LCS.	1) Internal standard recovery within limits stated in Table 2.	1) Check chromatography for interferences. If found, flag data. 2) Check S/N. If < 10:1, re-extract sample. 3) If S/N > 10:1, evaluate data usability, flag, narrate and report. 4) Check instrument and re-analyze the extract if a problem is found and corrected. 5) Re-extract and re-analyze adversely affected samples.
Method blank	1 per analytical batch, not to exceed 20 field samples per matrix.	No target analyte concentrations above the reporting limit (RL). Exception: OCDD concentration in the method blank is allowed to be 5X the RL without narration. Note "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for positive totals in the method blank.	1) Re-analyze method blank if instrument carryover is suspected. 2) If still exceeds and analyte concentration in sample < RL or > 10X blank concentration, narrate and report results. 3) If "J" qualified positives are in the method blank or OCDD < 5X the RL, then no corrective action is necessary. Flag and report 4) If non-compliant and analyte concentration in sample is between RL and 10X blank concentration, re-extract and re-analyze affected samples.
Laboratory Control Sample	1 per analytical batch, not to exceed 20 field samples per matrix.	Refer to Table 2.	1) Review Internal Standards, as above. 2) Evaluate data for usability. 3) If sample results are ND and RL are met, no action is required – narrate and report. 4) If samples have positives > RL, re-extract and re-analyze affected samples for analytes outside the acceptance criteria.
Duplicates	As per client request.	Refer to Table 2 and Table 3.	1) Review data for usability. 2) Narrate outliers.
Matrix Spike	As per client request.	Refer to Table 2 and Table 3.	3) Review data for usability. 4) Narrate outliers.
Matrix Spike Duplicate	As per client request.	Refer to Table 2 and Table 3.	1) Review data for usability. 2) Narrate outliers.

TABLE 5 CRITERIA FOR ISOTOPIC RATIO MEASUREMENT FOR PCDDs AND PCDFs Method 8290 – TestAmerica

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits (± 15%)
4	M/(M+2)	0.77	0.65-0.89
5	(M+2)/(M+4)	1.55	1.32-1.78
6	(M+2)/(M+4)	1.24	1.05-1.43
6[a]	M/(M+2)	0.51	0.43-0.59
7[b]	M/(M+2)	0.44	0.37-0.51
7	(M+2)/(M+4)	1.04	0.88-1.20
8	(M+2)/(M+4)	0.89	0.76-1.02

[a] Used only for ^{13}C-HxCDF (Internal Standard)
[b] Used only for ^{13}C-HpCDF (Internal Standard)

TABLE 6 PCDDs/PCDFs TOXICITY EQUIVALENCE FACTORS (TEF) Method 8290 – TestAmerica

Analyte	TEF March 1989 (EPA 62/5-89/016)	TEF June 1998 WHO	TEF June 2005 WHO
Dioxins			
2,3,7,8-TCDD	1.0	1.0	1.0
1,2,3,7,8-PeCDD	0.5	1.0	1.0
1,2,3,4,7,8-HxCDD	0.1	0.1	0.1
1,2,3,6,7,8-HxCDD	0.1	0.1	0.1
1,2,3,7,8,9-HxCDD	0.1	0.1	0.1
1,2,3,4,6,7,8-HpCDD	0.01	0.01	0.01
OCDD	0.001	0.0001	0.0003
Furans			
2,3,7,8-TCDF	0.1	0.1	0.1
1,2,3,7,8-PeCDF	0.05	0.05	0.03
2,3,4,7,8-PeCDF	0.5	0.5	0.3
1,2,3,4,7,8-HxCDF	0.1	0.1	0.1
1,2,3,6,7,8-HxCDF	0.1	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1	0.1
1,2,3,4,6,7,8-HpCDF	0.01	0.01	0.01
1,2,3,4,7,8,9-HpCDF	0.01	0.01	0.01
OCDF	0.001	0.0001	0.0003

TABLE 8 PCDDs/PCDFs HOLDING TIMES AND CONTAINERS
Method 8290 – TestAmerica

Method	**Extraction Holding Time**	**Containers** (no preservative other than 4°C)
8290	30 Days for soil and water	4 oz jar for soil; 2x 1 Liter amber for water